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FUNDAMENTAL RADIATION DAMAGE PROCESSES IN SILICON(U)  
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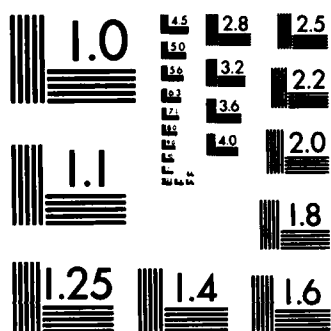
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October 25, 1983

Dr. Larry H. Cooper, Scientific Officer  
Code 427, Electronic and Solid State Science Program  
Office of Naval Research  
Arlington, Virginia 22217

REF: Closeout of Contract #N00014-76-C-1097  
"Fundamental Radiation Damage Processes in Silicon"

Dear Dr. Cooper:

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Sincerely,

*C. G. Roddy*  
C. G. Roddy  
Assistant Director

CC: G. D. Watkins  
W. B. Fowler

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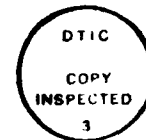
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## FINAL REPORT

### Fundamental Radiation Damage Processes in Silicon

N00014-76C-1097

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#### I. Introduction

The research on contract N00014-76-C-1097 was initiated September 15, 1976, and terminated September 30, 1983. In this final report, we summarize the work carried out during this period.

#### II. General Description of the Research Program

→ This program has been aimed at determining and understanding the fundamental properties of simple lattice point defects in semiconductors. The specific purpose has been to determine the electronic properties of the defects, to understand the mechanisms by which they are formed or incorporated into the lattice, and to probe the processes by which they can migrate through the lattice and react with other defects to form complexes. The major emphasis has centered on silicon. As the simplest and best understood semiconductor, silicon provides the best hope of clean, unambiguous and generally applicable answers to these questions. ←

In this study, the primary technique for producing defects has been high energy electron irradiation (1-3 MeV). Irradiation in situ at cryogenic temperatures has been performed as necessary to freeze in the simple primary lattice defects for study and to be able to separate the production mechanisms from the complex defect reactions when migration and annealing take place. Impurities have been incorporated by introduction in crystal growth, or by diffusion, or ion implant.

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The principal experimental techniques for studying the defects have been deep level transient capacitance spectroscopy (DLTS) and electron paramagnetic resonance (EPR). The EPR and DLTS studies have been combined in a coordinated attack to determine the electronic properties of the defects and their mechanisms of formation, migration, and reactions with other defects.

A closely interacting theoretical effort has also been incorporated into the program to investigate the electronic structure of deep level defects of interest to the experimental program. The approach has been to simulate a defect and its environment using a finite cluster of atoms and to calculate the electronic structure and to explore lattice relaxations around the defect using different quantum mechanical methods (SWX- $\alpha$ , MNDO).

### III. Summary of the Major Accomplishments Under This Contract

1. Negative-U defects. A defect has negative-U properties if it binds an electron (or hole) and then a second one, the binding energy of the second being greater than the first. It's as if there were a net attractive energy between the two charge carriers. This unexpected property, first suggested by Anderson<sup>1</sup> and extended by Street and Mott<sup>2</sup> to explain unusual properties observed for chalcogenide glasses implies an inversion of the normal order of levels in the forbidden gap for the defect. During this last contract period two simple point defects in silicon--the lattice vacancy and interstitial boron--have been demonstrated to have this remarkable property.<sup>3-11</sup> They constitute the first and only defects in any material to be unambiguously identified with negative-U properties.

Interstitial boron has been identified to have an acceptor level at  $E_c - 0.45\text{eV}$  and a donor level at  $E_c - 0.13\text{eV}$ . The negative-U for these inverted levels is therefore  $0.32\text{eV}$ . The proof of this involved a combination of EPR, DLTS, and a unique photo-DLTS experiment.

The lattice vacancy in silicon has been determined to have a negative-U of 0.08eV with a single donor state at  $E_V + 0.05\text{eV}$ , below the double donor level at  $E_V + 0.13\text{eV}$ . This proof was accomplished by a difficult and novel transient-EPR study using low energy (1) 300K black body radiation, (2)  $\text{CO}_2$  laser excitation, and (3) 20-25K thermal excitation to selectively populate and depopulate the hole states. A key to the success of this experiment was the availability of very low strain aluminum-doped silicon crystals, grown especially for us by H. Zuhlechner of Wacker Chemitronic, which allowed EPR observation of the aluminum acceptor resonance (normally too strain-broadened to be observed) so that the hole population could be continuously monitored between all of the relevant hole traps.

In the case of interstitial boron, the EPR experiments indicate that the driving force for the negative-U behavior is the large energy gained by a change in the bonding configuration for the defect as it traps the second electron. This therefore appears to be an example of the "valence alternation pair" model suggested by Kastner et al.<sup>12</sup> for such behavior. For the lattice vacancy, the EPR studies identify the origin as the large Jahn-Teller relaxations of the defect as the broken bonds reconstruct by pairs. This therefore provides an example similar to the mechanism originally proposed by Anderson.<sup>1</sup>

We consider these first observations of negative-U defects as extremely important: (i) They demonstrate for the first time that the phenomenon is physically possible and that it can indeed exist in real materials. (ii) The particular defects studied serve as model systems for understanding the mechanisms for the phenomenon. (iii) These studies provide the first insights into the possible role of negative-U defects in other semiconducting and insulating materials.

2. Recombination-enhanced migration and defect reactions. The phenomenon of enhanced diffusion of defects in semiconductors under electronic excitation is well documented.<sup>13,14</sup> This phenomenon is believed to be of practical importance in compound semiconductor devices which often degrade under operating injection conditions.<sup>13,14</sup> Unfortunately, none of the defects in these materials have been identified. In silicon, however, we have discovered as part of this research enhanced migration for several well characterized and identified defects, providing for the first time an opportunity to probe the fundamental microscopic processes involved in the phenomenon in this model system.

Detailed studies have been performed on interstitial aluminum, interstitial boron, and the lattice vacancy, all simple point defects produced by high energy electron irradiation at cryogenic temperatures.

a. Interstitial aluminum.<sup>15,16</sup> The second donor state was measured to be at  $E_V + 0.17\text{eV}$  by DLTS studies. By combining DLTS and EPR studies it has been established that the normal activation energy for migration is  $1.2 \pm 0.1\text{eV}$  in p-type material but that it can be reduced to  $0.27 \pm 0.03\text{eV}$  under minority carrier injection conditions. Normally stable to  $\sim 250^\circ\text{C}$ , the defect can be annealed therefore in a matter of minutes at room temperature under injection conditions. It was tentatively concluded that the mechanism was hole capture at the first donor state (estimated at  $\sim E_C - 0.2\text{eV}$ ) which releases  $\sim 0.9\text{eV}$  in energy to assist it over the barrier. This work has stimulated a recent flurry of activity among theorists at Bell Labs,<sup>17</sup> IBM,<sup>18</sup> MIT,<sup>19</sup> and in our group to try to model the system theoretically. This system is of particular fundamental interest because of its close chemical proximity to interstitial silicon which is known to migrate at  $< 4.2\text{K}$  under damage production conditions but about which very little else is known.<sup>20,21</sup>

b. Interstitial boron.<sup>4,16</sup> The normal activation energy for migration has been measured to be  $0.60 \pm 0.05 \text{ eV}$ . Under minority carrier injection conditions, the migration appears to be athermal, with no remaining energy barrier. The annealing rate displays an unusual quadratic dependence upon injection current in both n- and p-type material suggesting that the defect must cycle completely between the two stable  $B_1^+$  and  $B_1^-$  states in order to make a diffusional jump. This suggests a Bourgoin mechanism<sup>22</sup> in which the defect changes its lattice configuration as its charge state changes, one position being the saddle point for migration of the other and vice versa. This would agree nicely with the model developed for its negative-U behavior. However, we have also demonstrated that it is not sufficient to cycle from  $B_1^+ \rightarrow B_1^- \rightarrow B_1^+$  simply by electron capture and emission processes. Hole capture is necessary. Therefore a barrier apparently still remains that must be overcome by the extra energy of hole capture and a modified Bourgoin mechanism that includes such a barrier seems to be necessary.

c. Vacancy. The activation energy for migration of the vacancy depends strongly on its charge state and has been measured to be  $0.33 \text{ eV}$  in zero bias p-type,  $0.45 \text{ eV}$  in reverse bias p-type, and  $0.18 \text{ eV}$  in zero bias n-type. These presumably reflect the properties of  $V^{++}$ ,  $V^0$ , and  $V^-$ , respectively. Under injection conditions the migration is athermal, in n- and p-type material, with no remaining activation barrier. Detailed studies by both EPR and DLTS reveal a highly efficient process (under NdYAG laser excitation the vacancy makes one jump for every  $\sim 30$  photons incident on the sample). Several independent experiments indicate that the vacancy is positively charged when making its jump. This has been interpreted to mean that the energy release derives from the Jahn-Teller instabilities of  $V^+$  and  $V^0$ , the same electron-lattice coupling that provides its negative-U



behavior. Again, however, it is not sufficient to cycle between  $V^{++}$ ,  $V^+$ , and  $V^0$  simply by hole capture and emission processes. Again, electron-hole recombination, with its greater energy release to the defect, is required.

Whether the enhanced migration results from an energy release mechanism into one of the ground states, or motion over an excited energy surface available only from electron capture is not known at this stage. In either case, there remains the interesting question as to how the energy gets funneled into the trigonal diffusive mode, when the ground states of these charge states are observed to be tetragonally distorted.

d. Miscellaneous. The vacancy trapped next to a substitutional Ge impurity can still be electronically excited to migrate, and break-up of the pairs has been demonstrated. (This defect also appears to retain the negative-U behavior of the isolated vacancy). All other identified vacancy-defect pairs, however, have been found to be stable under electronic excitation (and possess positive-U properties). In several documented cases we have found that the vacancy appears to lose its ability to migrate under excitation as it gets near to a defect but before it reaches its final near-neighbor configuration. Apparently the lowering of the symmetry by the strain field of the other defect is sufficient to reduce the necessary structural flexibility of the vacancy.

Several other defects produced by electron irradiation at 4.2-20.4K have also been observed to anneal athermally under electronic excitation in EPR and DLTS. They have not been identified but there is evidence that some may be interstitial related.

3. Silicon self-interstitial studies. The growth of trapped interstitials reflects the release, migration and trapping of the self-interstitial, about which no direct information is as yet available. In p-type material,

this has apparently already occurred during the irradiation at  $\sim 4.2\text{K}$ . In n-type material, however, this may not be the case. The emergence of a DLTS level at  $E_c - 0.09\text{eV}$ , previously identified as arising from interstitial carbon,<sup>23</sup> has been studied vs. annealing after 3 MeV electron irradiation in situ at 4.2K. The study which included thermal annealing, charge state effects, and ionization enhancement has led to the following conclusions: The self-interstitial is released in two stages at  $\sim 150\text{K}$  and  $\sim 175\text{K}$ , corresponding to the loss of two EPR spectra, Si-L1, and Si-G25. In these stages the interstitial can be trapped by carbon, forming the  $E_c - 0.09\text{eV}$  level, or by oxygen. The interstitial is subsequently released by oxygen at  $\sim 250\text{K}$ , forming more interstitial carbon. Charge state effects indicate that the self-interstitial is positively charged when migrating in n-type silicon.

4. Transition elements in silicon. EPR studies have been initiated to probe the structure and electrical properties of transition elements in silicon. One study, recently completed, involves  $\text{Pt}^-$  in silicon. Henning and Egelseers<sup>24</sup> recently concluded that the EPR center first observed by Woodbury and Ludwig<sup>25</sup> and identified by them as isolated substitutional  $\text{Pt}^-$  in silicon was really a Pt-Pt pair. We have selectively doped silicon with  $\text{Pt}^{198}$  and  $\text{Pt}^{195}$  and have shown conclusively that the "satellites" seen by Henning et al. do not reveal the presence of a second Pt at all and the original isolated  $\text{Pt}^-$  model of Ludwig and Woodbury is correct. It was important to challenge the pair model and prove it wrong because the  $\text{Pt}^-$  spectrum plays an important role in understanding the "vacancy-like" character of substitutional impurity ions.<sup>26</sup>

5. Theory of deep levels. A systematic study of the interstitial 3d transition element ions in silicon, using finite clusters of atoms to simulate the crystalline silicon environment and scattered wave X- $\alpha$  quantum

mechanical techniques has been carried out.<sup>27-30</sup> Many-electron effects have been estimated both by spin unrestricted calculations and by using the Hemstreet-Dimmock<sup>31</sup> approximation. The experimentally observed high spin Hund's rule states have been correctly predicted in all cases. Many-electron corrections were applied (for the first time in any defect system) to the predicted level positions with reasonable agreement with experiment. The critical ions for testing the various approximations in the theory (Fe for the magnitude of the crystal field splitting and V for the relative magnitude of crystal field and exchange effects) have been identified.

Another important theoretical problem for which studies have been undertaken is that of off-center substitutional first row element impurities (O,N) in silicon. The mechanism for this distortion (Jahn-Teller, pseudo-Jahn-Teller, molecular bonding, etc.) is a subject of current controversy. It is an important question because similar effects are believed to be occurring in the compound semiconductors as well. For this study, we have again used a cluster approach but the quantum mechanical methods have been augmented with the MNDO technique which supplies total energies. The correct off-centered distortions can be reproduced within certain restrictions for the terminations of the clusters. Our results have tentatively identified the mechanisms for the distortions as of pseudo-Jahn-Teller origin.<sup>30,32</sup>

The SWX- $\alpha$  and MNDO techniques have also been applied to the interaction between hydrogen or alkali atoms and a silicon lattice vacancy.<sup>33</sup> As expected, hydrogen passivates the dangling bonds, with four in a vacancy required to completely passivate the defect. A surprise, however, is that lithium instead appears to move into the nearby interstitial site as  $\text{Li}^+$  and donates its electron to the vacancy. We predict that four  $\text{Li}^+$  ions

tetrahedrally surrounding a  $V^{4-}$  vacancy would form a stable deep multiple donor.

6. Fano Resonances in the Optical Excitation Spectra of Acceptors in Silicon. We have detected and analyzed dips in the excitation spectra for Group III acceptors in silicon as resulting from Breit-Wigner-Fano resonance interactions between the electronic continuum states of the acceptors and localized excitations involving the  $519\text{ cm}^{-1}$  zone center phonon. This is the first time these interactions have been observed directly in excitation spectra.

#### IV. Publications Resulting from This Contract

##### a. Journal articles

1. G. D. Watkins, "Displacement Defects in Semiconductors Produced by Electrons," in Proceedings of the Fourth Conference on the Scientific and Industrial Applications of Small Accelerators, (IEEE, 76 CH. 1175-9NPS) edited by J. L. Duggan and I. L. Morgan, p. 588.
2. G. D. Watkins, "Lattice Vacancies and Interstitials in Silicon," Chinese Journal of Physics 15, 92 (1977).
3. G. D. Watkins and W. Beall Fowler, "Resonant interactions of optical phonons with acceptor continuum states in silicon," Phys. Rev. 16, 4524 (1977).
4. G.D. Watkins, J. R. Troxell, and A. P. Chatterjee, "Vacancies and Interstitials in Semiconductors," in Defects and Radiation Effects, 1978, edited by J. H. Albany (Inst. of London, Conference Series 46, 1978), p. 16.
5. J. R. Troxell, A. P. Chatterjee, G. D. Watkins, and L. C. Kimerling, "Recombination-enhanced migration of interstitial aluminum in silicon," Phys. Rev. 19, 5336 (1979).
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12. G. D. Watkins, "Summary of the 1980 Conference," in Defects and Radiation Effects in Semiconductors 1980, edited by R. R. Hasiguti (Inst. Phys. Conf. Se. #59, 1981), p. 139.
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17. G. D. Watkins, "Anisotropic-Defect Production in Compound Semiconductors by Electron Irradiation," *Phys. Rev. Letters* 48, 568 (1982).
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20. G. D. Watkins, "Deep Levels in Semiconductors," *Physica* 117B/118B, 9 (1983).
21. G. D. Watkins, G. G. DeLeo, and W. B. Fowler, "Theory of Defects in Silicon: Recent Calculations Using Finite Molecular Clusters," *Physica* 116B, 28 (1983).
22. J. L. Newton, A. P. Chatterjee, R. D. Harris, and G. D. Watkins, "Negative-U Properties of the Lattice Vacancy in Silicon," *Physica* 116B, 219 (1983).
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24. G. D. Watkins, "The Lattice Vacancy in Silicon," in Deep Level Impurities, edited by S. Pantiledes (Gordon and Breach, to be published).
25. G. G. DeLeo, W. B. Fowler, and G. D. Watkins, "Electronic Structure of Hydrogen- and Alkali-Vacancy Complexes in Silicon," submitted to *Phys. Rev. B*.
26. G. G. DeLeo, W. B. Fowler and G. D. Watkins, "Theory of Off-Center Impurities in Silicon: Substitutional Nitrogen and Oxygen," submitted to *Phys. Rev. B*.
27. R. F. Milligan, F. G. Anderson, and G. D. Watkins, "Electron Paramagnetic Resonance of Pt<sup>-</sup> in Silicon: Isolated Substitutional Pt vs. Pt-Pt Pairs," submitted to *Phys. Rev. B*.

b. Presentations at Scientific Meetings

A total of thirty-nine papers were presented at scientific meetings on work related to this contract during this period. Of these eighteen were invited papers.

c. Theses Completed

1. J. R. Troxell, "DLTS Studies of Radiation Induced Defects in Silicon," PhD dissertation, Lehigh University, 1979.
2. R. D. Harris, "A Study of Defects Produced in Silicon by Electron Irradiation at Cryogenic Temperatures," PhD dissertation, Lehigh University, 1982.
3. A. P. Chatterjee, "Ionization-Enhanced Migration of Defects Produced in Silicon at Cryogenic Temperatures," PhD dissertation, Lehigh University, 1982.

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